Measurement of Service Area for Television Broadcasting

A NEW METHOD of measuring the service provided by a television broadcast station has been devised at the Boulder Laboratories of the National Bureau of Standards. This method involves an area concept of service rather than the contour concept presently being used. In current practice, television service is defined in terms of the contours for two grades of service, grades A and B. This definition presents two difficulties. First, the contour does not adequately express service because it includes within it many locations that receive less than the specified grade of service and ignores large areas outside the contour that have the specified grade or better. Second, the experimental determination of where the contours lie is very difficult to accomplish adequately.

A more proper definition of service was first proposed by Norton and Gainen [1] in 1951. This definition expresses the service in terms of the total area that receives service in various grades. The grades of service are related to the signal levels, or more properly, to the signal-to-noise ratios. Grade I service would represent the cases where the signal is sufficiently high to override thermal and manmade noise levels; grade II would represent a lower signal-to-noise ratio where perhaps noise is somewhat noticeable; and grade III the case where the signal-to-noise level is low and a considerable amount of noise appears at reception.

Because of terrain roughness, including trees, buildings, etc., the distribution of the actual areas is not uniform. It is entirely possible for one dwelling to receive grade I service and for next-door neighbors to receive grade II or even grade III. Within roughly 50 miles of the transmitter, time variations are relatively unimportant; but as the distance increases, tropospheric effects become more important. An expression of the time available must be included in the definition of service.

Current practice in measuring the contours of service involves recorder-tape measurements of field strength. These measurements are made in moving vehicles while driving down roads along eight or more radials from the transmitter. From these measurements the contours, where 50 percent of the locations receive the specified grade of service for 50 percent of the time, are estimated and joined together in the form of a service contour.

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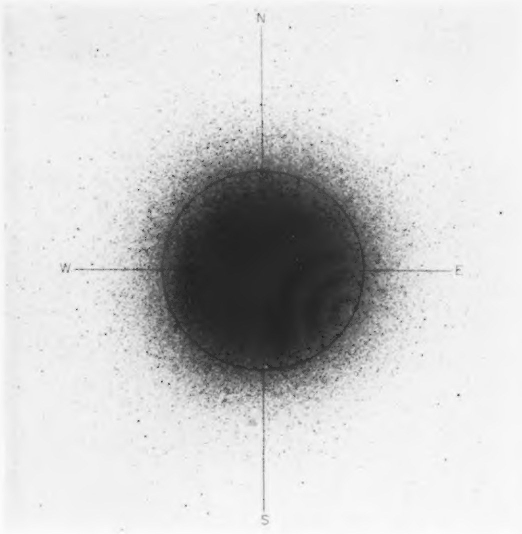
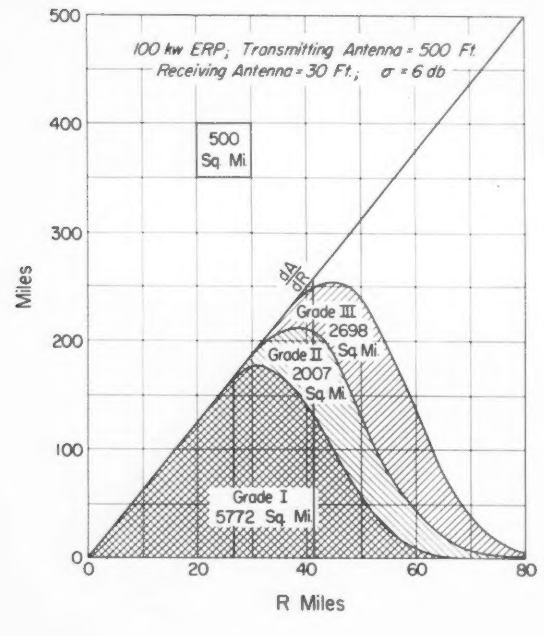




MINIMUM FIELD STRENGTH AND PERCENTAGE OF TIME AVAILABILITY DEFINING THREE GRADES OF TELEVISION BROADCAST SERVICE AT 63 Mc (FROM NORTON AND GAINEN)

	T_a%	URBAN $p \geq 5000$	SUBURBAN $150 < p < 5000$	RURAL $p < 150$
GRADE I	99	77	67	57
GRADE II	90	72	62	52
GRADE III	50	67	57	47

Aside from the fact that the contours do not give a very useful representation of service, there are many difficulties in taking measurements in this manner. First of all, it is impossible to obtain very much of this type of data using the proper height of antenna, namely, 30 feet. Most such measurements are made at 10 feet and referred to the 30-foot reference height by application of a linear height-gain function. Secondly, the routes followed usually do not represent the area very well because (1) roads tend to follow lower levels in rough terrain, (2) they are frequently built up a few feet above surrounding terrain, and (3) they are congested with wires and other objects which modify the results. Time and space variations cannot be separated in mobile measurements.

A method of measurement that will lead to an efficient estimate of the areas of service is proposed by the Bureau to replace the old method. In this method sample measurements are taken at fixed locations around the transmitter. These locations are established in a systematic manner so that all terrain types are equally likely and the data represent a random sample from the area. In most cases the measurements consist of a single observation of field strength except when time variations are involved. In these instances they represent a short recording of field strength from which the distribution with time can be estimated. A sample set of observations would be taken at a constant radius with sufficient separation to eliminate serial correlation between successive observations. The distribution obtained can be used to estimate the percentage of locations at the distance that receives service in the several grades. By joining together several such estimates made at different distances, an estimate can then be made of the total area around the transmitter for each grade of service.

Some of the advantages of this method over mobile measurements are as follows:

1. Much better estimates of the population parameters are obtained.

2. Proper account is taken of the statistical nature of the received field, in particular, serial correlation.

Above: **Diagram illustrating how a given minimum grade of service might be distributed around a television transmitter under average conditions. In black areas the field strength exceeds the prescribed level, in white areas it falls short of this value. The circle illustrates how a single contour is used in the current method to represent service. This tends to ignore large areas outside the contour that have the required field strength, and to include areas within the contour that do not. The actual service area would be the sum of all black areas.** ***Center:*** **Hypothetical television service areas for channels 2 to 6 based on FCC propagation curves. The curves shown are based on rural values of field strength from tabulation at bottom. Field strength is expressed in decibels above 1 μv m at a point 30 ft above the ground.**

3. The measurements can easily be obtained at a height of 30 feet and at locations more typical of potential receiver locations.

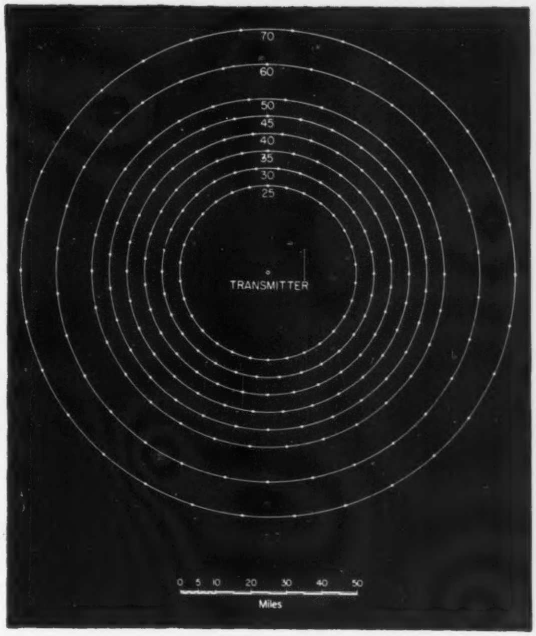
4. Time variations can be measured separately from space variations.

5. The analysis is facilitated by the elimination of long recorder tapes. All of the scaling and some of the analysis can be done in the field. In most cases the tabulation consists of writing down a single value read directly from the instrument, and in others it involves at most the determinaion of the distribution with time from a relatively short observation. The tabulated values then might be, for example, the field strength exceeded 99, 90, and 50 percent of the time.

6. The measurements are more suitable for technical study and analysis and are repeatable in the sense that repetitive sampling from the same parent population gives estimates where dispersion is expressed by a confidence interval.

7. Additional studies can easily be incorporated into the measurements. Such studies might include cross-polarization effects; direction of arrival measurements, path antenna gain, and many others.

[1] A study of the geographical distributioa of population on the coverage obtainable from a national television system, *by K. A. Norton and L. Gainen, a working document of the Federal Communications Commission Ad Hoc Committee for the Evaluation of the Radio Propagation Factors Concerning the Television and Frequency Modulation Broadcasting Services in the Frequency Range Between 50 and 250 Mc, October 17, 1950.*

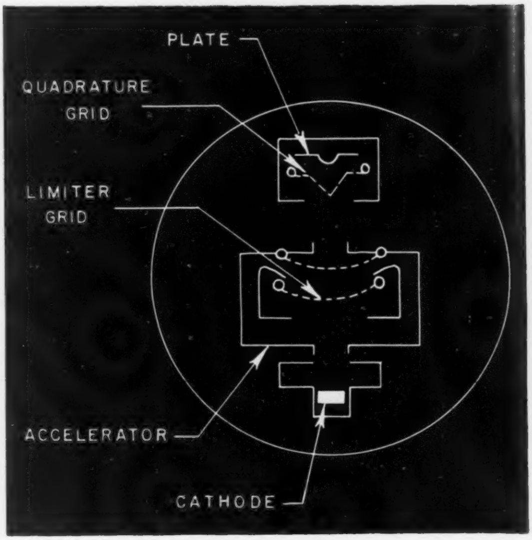


Above: **One possible plan for sampling the field intensity distribution of a 100 kw television station over average terrain, according to a method worked out by the Bureau. In this case a total of 240 measurements would be made, consisting of 30 discrete observations on each of 8 circles with radii from 25 to 70 miles, respectively.** ***Below:*** **Photographs illustrating two ways in which sample measurements of television field strength have been obtained by Bureau scientists. When it is impossible to drive to specific locations, a small battery-operated field strength meter (right) can be packed to the spot. Measurements at antenna heights of both 15 and 30 ft showed clearly that the simple linear height-gain function currently in use to extrapolate low-antenna measurements to a height of 30 ft is not satisfactory, especially in very rough terrain.**

A SIMPLIFIED PULSE GENERATOR

A SIMPLIFIED, inexpensive circuit for a relaxation oscillator with fast rise and decay times has been developed by the Bureau's sound laboratory.[1] The special advantages of the circuit are due mainly to the gated-beam tube, type 6BN6, around which it is designed. Thus, in comparison with the conventional blocking oscillator or multivibrator, whose basic circuits are similar to the present one, use of the gated-beam tube permits the elimination of a transformer or an additional tube for positive feedback. The only components required, besides the gated-beam tube itself, are 3 or 4 resistors and 1 condenser. C. E. Tschiegg of the NBS sound laboratory designed the pulse generator, and the work was supported in part by the Office of Naval Research.

The principle of operation resembles that of the Van der Pol relaxation oscillator,[2] but again important advantages result from the gated-beam tube. Pulse widths ranging from about 0.5 to 400 μsec can be obtained—and at frequencies greater than 1 Mc for the narrow pulses. With low impedance load, pulse rise and decay times can be made less than 0.1 μsec, even for pulse durations as large as 400 μsec. In addition, use of the gated-beam tube makes possible very simple biasing and triggering arrangements.



Schematic cross section of gated-beam tube used in relaxation oscillator. The boxlike accelerator electrode shields the cathode from the other electrodes, so that the cathode current is held essentially constant. The potentials on the limiter and quadrature control grids determine how the current is shared between accelerator and plate. If limiter is sufficiently negative, electrons are repelled to the inside wall of the accelerator; and if the quadrature grid is too negative, electrons are repelled to the outside of the accelerator. Either grid can cut off the plate current, regardless of potential on other grid.

The Gated-Beam Tube

Originally designed as a limiter-discriminator for TV and FM receivers, the gated-beam tube is a pentode with a construction rather different from that of most five-element tubes. Electrons from the filament, formed into a vertical sheet by a slit, first pass through a boxlike electrode, the "accelerator," which has openings front and back for the electron beam. Of the two control grids, the "limiter," is within the accelerator enclosure; and the "quadrature" grid, is between the accelerator and the plate.

The accelerator effectively shields the cathode from the other electrodes, so that the cathode current is essentially constant. The control grids serve as gates to distribute this current between the normally positive accelerator and plate. If the limiter is sufficiently negative, its shape is such that the electrons are deflected onto the inner surface of the accelerator, instead of passing through. Similarly, when the quadrature grid is sufficiently negative, it repels electrons onto the outer surface of the accelerator. As a result, the plate current can be cut off completely by either of the two control grids, regardless of the potential on the other one.

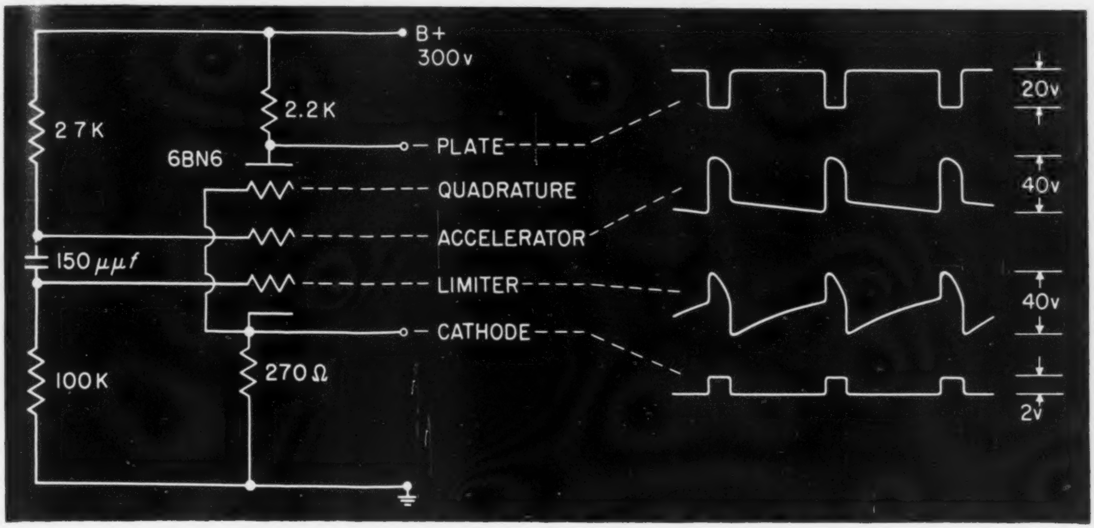
In addition, it is possible to self-bias the tube to plate-current cutoff with a resistor in the cathode circuit. This feature, together with the availability of a second grid for high impedance trigger, makes the gated-beam tube exceptionally well suited for use in triggered oscillators.

Oscillator Operation

With appropriate values for the components, gated-beam oscillators of either free-running or triggered type can be constructed. An example of a free-running oscillator is given in the accompanying schematic diagram. Here each pulse corresponds to a cycle of operations in which the current is switched from the accelerator to the plate and back.

Consider an instant when the plate current (in the circuit illustrated) is cut off by a negative potential on the limiter. This condition is unstable, because a current flows through the resistor connecting the limiter to ground, discharging the condenser and raising

Schematic diagram of free-running pulse generator together with the voltage waveforms (right) produced at the tube electrodes. Taking advantage of the special characteristics of the gated-beam tube, this circuit eliminates the transformer or additional tube for positive feedback required in the somewhat similar blocking oscillator or multivibrator circuits. It generates pulses from 0.5 to 400 μsec in width, with pulse rise and decay times below 0.1 μsec; the narrower pulses can be generated at frequencies above 1 Mc. Cathode resistor can be removed if output is taken from plate, or it can be changed from 270 to 560 ohms to produce a triggered oscillator. Gated-beam tube makes possible simple biasing and triggering arrangements.



the limiter potential. The circuit remains quiescent, however, until the limiter potential rises sufficiently to permit electrons to reach the plate. When this happens—that is, when the limiter reaches cutoff and plate current begins to flow—there is a corresponding decrease in accelerator current and increase in accelerator potential. The potential increase is transmitted through the condenser and reinforces the plate current. Because of this regenerative process, the plate current very rapidly rises to its maximum value (when most of the accelerator current has been diverted to it).

During the regenerative process, the limiter is driven positive and draws current, enabling the condenser to charge rapidly. When maximum plate current is reached, ending the regenerative process, the charge on the condenser drives the limiter negative. This cuts off the plate current and the cycle starts again.

By increasing the cathode resistor from 270 to 560 ohms, the circuit described can be converted to a triggered oscillator. The potential drop across this resistor makes the limiter sufficiently negative with respect to cathode to cut off the plate current. If a positive trigger is now inserted into the limiter (or a negative trigger into the cathode), the oscillator goes through a single cycle of the kind described above. It then returns to its original condition and is held there by the cathode bias until the next trigger pulse arrives.

The PRF of the free-running oscillator, or the recovery time in the triggered oscillator, is controlled by the time constant of the R–C combination connecting the accelerator to ground. The pulse width depends on the capacity of the condenser and the internal impedance of the limiter grid. Greater output can be obtained by increasing the plate resistor, but this also retards the rise and decay of the pulses. In the free-running oscillator, the cathode resistor can be eliminated if the output is taken from the plate.

Among possible modifications of the circuit is the use of an inductive plate load or a tuned circuit in the feedback path. A series resonant crystal could also be used, provided some means for limiting the crystal current is included. The PRF will then be the same as the fundamental or some subharmonic [3] of the crystal frequency, depending on the time constant that controls the unsynchronized PRF. A tuned circuit can be used in the output if a sinusoidal waveform is desired.

[1] Relaxation oscillator using a gated-beam tube, *by C. E. Tschiegg, Rev. Sci. Instr.* **27**, 1085 (*December 1956*). (*In the circuit diagram accompanying this article, the connections to the limiter and quadrature grids should be interchanged.*)

[2] On "relaxation oscillators," *by B. van der Pol, Phil. Mag.* **2**, 978 (1926).

[3] Subharmonic crystal oscillators, *by M. C. Thompson, C. E. Tschiegg, and M. Greenspan, Rev. Sci. Instr.* **25**, 8 (1954).

Crystallography of Cold Drawn Music Wire

THE BUREAU has completed an investigation of preferred crystal orientation in cold drawn music spring wire. Sponsored by the U. S. Army Ordnance Corps and conducted by H. C. Burnett and C. J. Newton of the Bureau's mechanical metallurgy laboratories, the study revealed that the degree of preferred crystal orientation and the elastic modulus of the wire first increase, then decrease, as diameter is reduced in the drawing process. The tensile strength of the wire increases with each reduction in size, as expected, but exhibits no correlation with changes in either the preferred orientation or the modulus of elasticity.

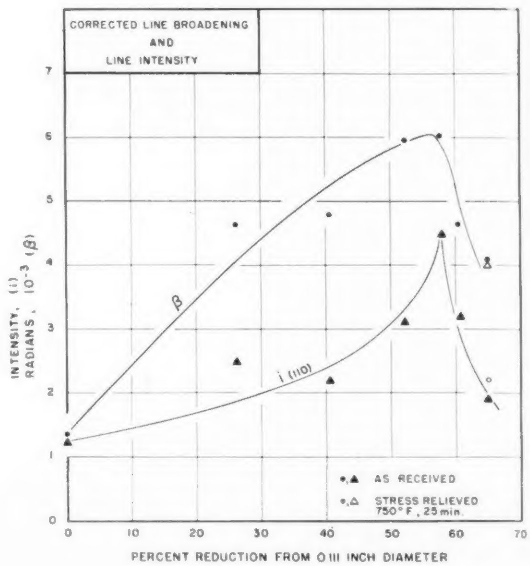
Many modern ordnance devices such as machine guns and small arms are dependent on helical wire springs for their operation. Music wire is most frequently used for these springs because it is the strongest economical engineering material available. The usual fabrication procedure for music spring wire involves a heat treatment known as patenting followed by drawing through a series of dies to obtain the desired size and tensile strength. The plastic deformation of the metal during the drawing operation causes the individual crystals to rotate so that their crystallographic axes tend toward certain preferred directions. As the properties of the crystals are different in different directions, the development of a preferred orientation in a metal specimen causes the properties of the specimen to vary with direction. It was believed that information on changes in preferred orientation and elastic modulus occurring during wire drawing would aid in obtaining a better understanding of the structural changes taking place in the steel.

Commercial-grade wire drawn from steel of 0.85 percent carbon content was used in the investigation. The patented wire had a diameter of 0.111 in., and 11 dies were used in reducing the wire to the final size of 0.039 in.

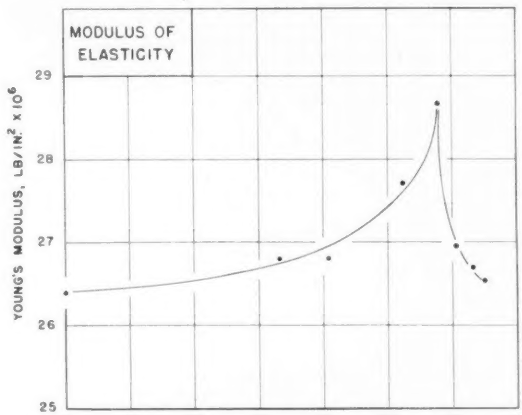
The degree of preferred orientation was determined on seven sizes of wire and on an 0.039-in.-diameter specimen which was stress-relieved by heating at 750° F. The preferred orientation data were taken with an X-ray diffractometer and associated counting, scaling, and recording circuits. The radiation source was cobalt K-alpha.

In order to obtain sufficient area for use in the diffractometer, a number of short lengths of wire were cemented together and then mounted in Bakelite for cutting and polishing. The surface of each specimen was cut at some specific angle with respect to the axes of the wires, metallographically polished, etched to remove the cold worked layer resulting from the cutting operation, and then lightly polished.

The results showed that the preferred orientation increased with increased reduction of area by drawing until a diameter of 0.047 in. was reached. Further drawing caused a sharp decrease in the degree of preferred orientation so that the value for the 0.039 in.



***Above:* Graph of corrected line broadening and intensity of the diffraction from the (110) plane of music spring wire at various stages of reduction. Specimens were cut transversely across the axis of the wire. *Below:* Graph of modulus of elasticity of music wire at various stages of reduction.**



wire was only slightly greater than that of the patented material.

Young's modulus of elasticity was determined for 18-in. lengths of wire in the drawn condition at eight stages of reduction. The data were obtained by direct static loading in a vertical plane. Strain was meas-

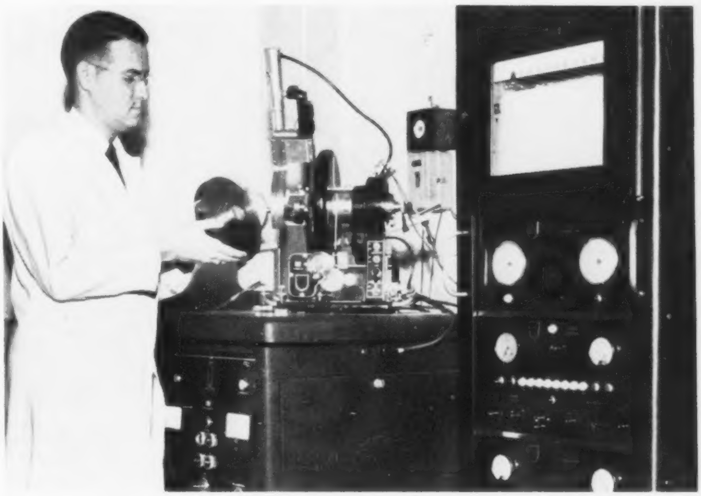
ured with two Tuckerman extensometers mounted opposite each other on the specimen. Mounting in this manner automatically compensated for any bending of the specimen during the initial loading. Ten to twelve additions of load were made on each wire being tested, and the increments of strain were obtained by averaging the reading of the two extensometers after each addition of load.

The value of Young's modulus was found to rise to a maximum with wire of 0.047-in. diameter and thereafter to decrease as did the preferred orientation. The development of preferred orientation, therefore, seems to be the principal factor contributing to the change of modulus.

For further technical information, see Variations in preferred orientation and modulus of elasticity in cold drawn music spring wire, *by H. C. Burnett and C. J. Newton, Wire and Wire Products,* **30**, *1375 (Nov. 1955)*.

TABLE 1. *Tensile strength of cold-drawn music wire at various stages of reduction*

Wire diameter	Reduction in diameter	Tensile strength
in.	%	*lb/in.²*
0.111	----	206,000
.101	9.0	229,000
.091	18.0	236,000
.082	25.1	244,000
.074	33.3	254,000
.066	40.5	264,000
.059	46.8	278,000
.053	53.2	292,000
.047	57.7	312,000
.044	60.2	322,000
.041	63.0	332,000
.039	64.8	350,000



Right: **Making X-ray diffraction measurements on samples of cold drawn music spring wire. The study showed how crystal orientation changes as the wire diameter is reduced in the drawing process.**

X-ray Attenuation Coefficients From 10 kev to 100 Mev

IN RESPONSE to widespread requests for basic input data for evaluating the penetration of radiation, the Bureau recently issued NBS Circular 583, *X-ray Attenuation Coefficients From 10 kev to 100 Mev.*[1] Some of the data presented has already been published in radiological handbooks and technical journals. However, the present publication is a more extensive and systematic study. It will be useful to physicists studying basic problems in radiation and nuclear physics, to physicians and hospitals utilizing radiation from X-ray generators and gamma rays from radioactive substances, and to designers of shielding for nuclear reactors.

In addition to tabular and graphical data for 29 absorbing materials, which make up the bulk of the publication, there is a discussion of the principal absorption and scattering processes involved in the interaction of radiation with matter; namely, photoelectric effect, scattering (Compton and Rayleigh), and pair production. Existing theoretical and experimental data for these processes are here compiled and evaluated.

Separate tabulations have been made for the probabilities of the dominant interactions as well as the total attenuation coefficient—the sum of the above processes. It appears that the present information is adequate for many applications. However, improved theory and additional experimental data are needed in certain areas. This need is pointed up by a comparison of the calculated and experimental coefficients.

[1] *Copies of the 54-page circular are available at 35 cents each from the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C.*

STANDARD MUSICAL PITCH

ONE of the lesser known services of the National Bureau of Standards is the broadcasting of a musical tone of standard pitch—middle "A" at 440 cycles per second—over its shortwave stations WWV (Beltsville, Md.) and WWVH (Maui, Hawaii). These broadcasts make standard pitch available day and night throughout the United States and over much of the world. Because a shortwave receiver is all that is needed, easy access to standard pitch is thus provided for piano tuners and amateur and professional musicians, as well as for makers of musical instruments.

A 600-c/s tone is also broadcast. This, together with the 440-c/s tone, is used by scientists, electronics engineers, and manufacturers in the measurement of short intervals of time and for calibrating instruments and devices that operate in the audio and ultrasonic frequency ranges. Both the 440- and the 600-c/s tones are obtained from an electronic, crystal-controlled oscillator and are accurate, as transmitted, to better than 1 part in 100,000,000.

The two frequencies are broadcast alternatively, starting with 600 c/s on the hour for 3 min, interrupted 2 min, followed by 440 c/s for 3 min and interrupted 2 min. Each 10-min period is the same except that WWV is off the air for 4 min beginning at 45 min after each hour; and WWVH is silent for a 34-min period each day beginning at 1900 Universal Time (9 A. M. in Hawaii or 2 P. M. EST).

To provide greater assurance of reliable reception, transmissions from the NBS stations are made simultaneously on several standard broadcast frequencies. WWV broadcasts on 2.5, 5, 10, 15, 20, and 25 Mc (megacycles per second) and WWVH broadcasts on 5, 10, and 15 Mc.

In this country, $A = 440$ c/s has been accepted as standard pitch since 1925. Initially, this value was agreed upon by the Music Industries Chamber of Commerce as a useful compromise among the various pitches chosen arbitrarily by different musical groups. In 1936 the same pitch standard was adopted by the American Standards Association, giving it the status of an industrial standard. Three years later the International Federation of National Standards Associations (ISA)[1] sponsored a conference in London. France, Germany, Great Britain, Holland, and Italy sent delegates, and the United States and Switzerland sent official messages. Six of the seven countries independently proposed $A = 440$ as the standard and the conference adopted it unanimously. The same standard was again endorsed by the International Organization for Standardization (ISO) in 1953; and was accepted as an ISO Recommendation at Stockholm in 1955.

The National Bureau of Standards maintains the $A = 440$ standard as the one on which general agreement has been reached. The musical merits of any particular standard are, of course, outside its province.

Earlier Pitch Standards

Previous standards of pitch[2] were defined in terms of the frequency of a particular tuning fork or bar, or the length of a specified vibrating air column (organ pipe). Because the sound frequencies generated by these devices vary with the surrounding temperature, it is necessary to specify the temperature at which comparisons with these standards should be made.

In 1859 the "Diapason Normal" was defined in terms of a standard tuning fork deposited by the French Government at the Paris Conservatory of Music. The vibration frequency of this fork was stated to be 435 c/s when measured at the then standard laboratory temperature of 15° C. When R. Koenig (1880) made a careful determination of the frequency, it proved to be 435.45 c/s at 15° C and to have a thermal coefficient of -0.0486 c/s per degree centigrade. Thus the fork would really have the defined standard frequency at slightly over 24° C.

An international congress in Vienna in 1891 adopted the French definition of the Diapason Normal, and it acquired the name of "International Pitch." Great Britain and the United States apparently did not at-

tend this meeting, though A=435 was used as a standard by a number of musical groups and instrument makers in this country after its adoption by the Vienna congress.

In many places the pitch standards in actual use were strongly influenced by the way large, permanently installed pipe organs were tuned. Yet, of all the mechanical devices used to generate musical frequencies, the vibrating air column of the pipe organ is the most sensitive to changes in temperature. Their frequency would therefore depend on what the temperature happened to be when they were adjusted to conform to the standard fork in the Conservatory at Paris. Since the advent of better heating systems and air conditioning, the temperature at which most musical instruments are used today—in the United States, at least—is better represented by 20° C (68° F) than by the temperature of 15° C (59° F) associated with the Diapason Normal. Luckily, an organ pipe tuned to A=435 at 15° C will actually be tuned almost exactly to A=440 at 20° C.

Advantages of Present Standard

From a technical point of view, the present standard of musical pitch, as maintained by the Bureau, has the advantage that it is free from the vagaries of the material objects (tuning forks, organ pipes) that embodied past standards. Thanks to modern electronic techniques for generating and stabilizing oscillations, a tone is produced that for all practical purposes is independent of the temperature of the surroundings.

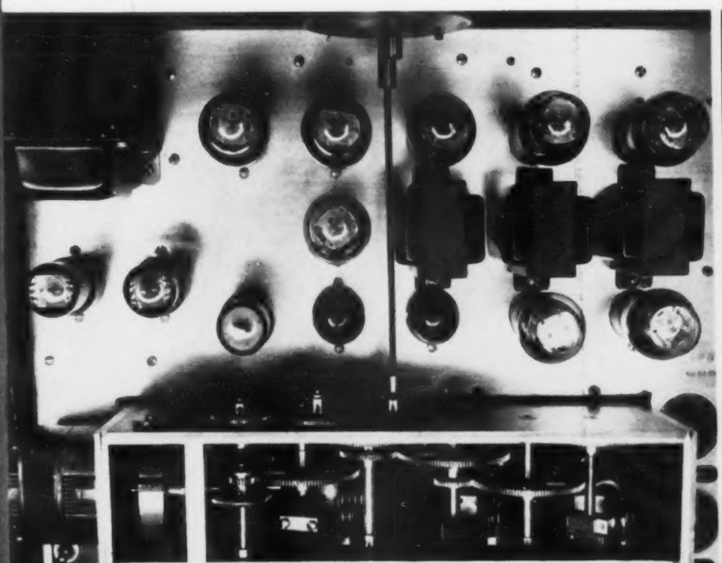
This would apply, of course, to any musical frequency that might be chosen. It happens, however, that the 440-c/s frequency stands in a very simple relation to other frequency standards maintained by the Bureau and can therefore be produced with a minimum of additional equipment. A tone of 435-c/s, for example, would require a somewhat more complicated technical arrangement.

In changing over to Standard Pitch, little or no alteration is necessary in adjusting instruments tuned to the older standard. Instruments tuned by string tension and the open vibrating air columns of pipe organs present no problems at all. Woodwinds can be corrected partly by the tuning adjustment of the instrument and partly by the breath control of the player: and changes required in the reed stops of the organ are within the range of the instrument's tuning adjustments.

[1] *This has now been superseded by the International Organization for Standardization (ISO).*

[2] *A history of earlier standards of pitch is appended to the English translation of* Sensations of Tone, *by Helmholtz, translated by A. Ellis, published by Longmans, Green & Co., New York, 1895 (reprinted by Dover, New York, 1954). A history of British standards of pitch is given in the folder,* British Concert Pitch, *published by British Standards Institution, 28 Victoria St., London, SW1.*

Below, left: Seconds-pulse generator and time-interval selector at station WWV. This serves as the motivating unit to remove (and then return) the audiofrequency tones—standard musical pitch at 440 c/s and a 600-c/s tone—from the broadcast signal for periods of 1/25 sec once each second. During the 1/25-sec interval a short train of 1000-c/s oscillations is used to provide a seconds "tick." The mechanical gears shown (below) serve only a gating purpose and do not determine the precision of the timing operations. The precision is electronically controlled. _Right:_ Audiofrequency racks used for generating standard musical pitch, 440 c/s (A above middle C), at station WWV. The day and night broadcasts from WWV and from the Bureau's other station, WWVH in Hawaii, make the standard tone available to shortwave sets throughout the United States and a large part of the world. The racks shown also generate a 600-c/s tone, and each of the two tones is broadcast for 3 min in alternate 5-min intervals. Three audioracks are maintained, two of them for standby purposes in case of failure of the one in use. The engineer is here adjusting the phase relationships of the audio oscillations generated by the different racks to keep them in accurate synchronism.



Cinchonine Analysis of Tanning Materials

THE BUREAU, in cooperation with the American Leather Chemists' Association, has developed a method of determining the amount of lignosulfonates present in tanning materials. The method is based on the precipitation of lignosulfonates with cinchonine. Tests made in six laboratories with 24 contrasting samples show that the method is generally applicable, that it is simple and fast, and requires no special equipment.[1]

Lignosulfonates, obtained as waste products in the manufacture of cellulose from wood, are often used in the tanning of leather. Possessing no tanning properties of their own, they act as auxiliary agents and as extenders for more costly vegetable tannins. However, large amounts of lignosulfonates in tanning materials become harmful adulterants, and a method for their control has long been sought.

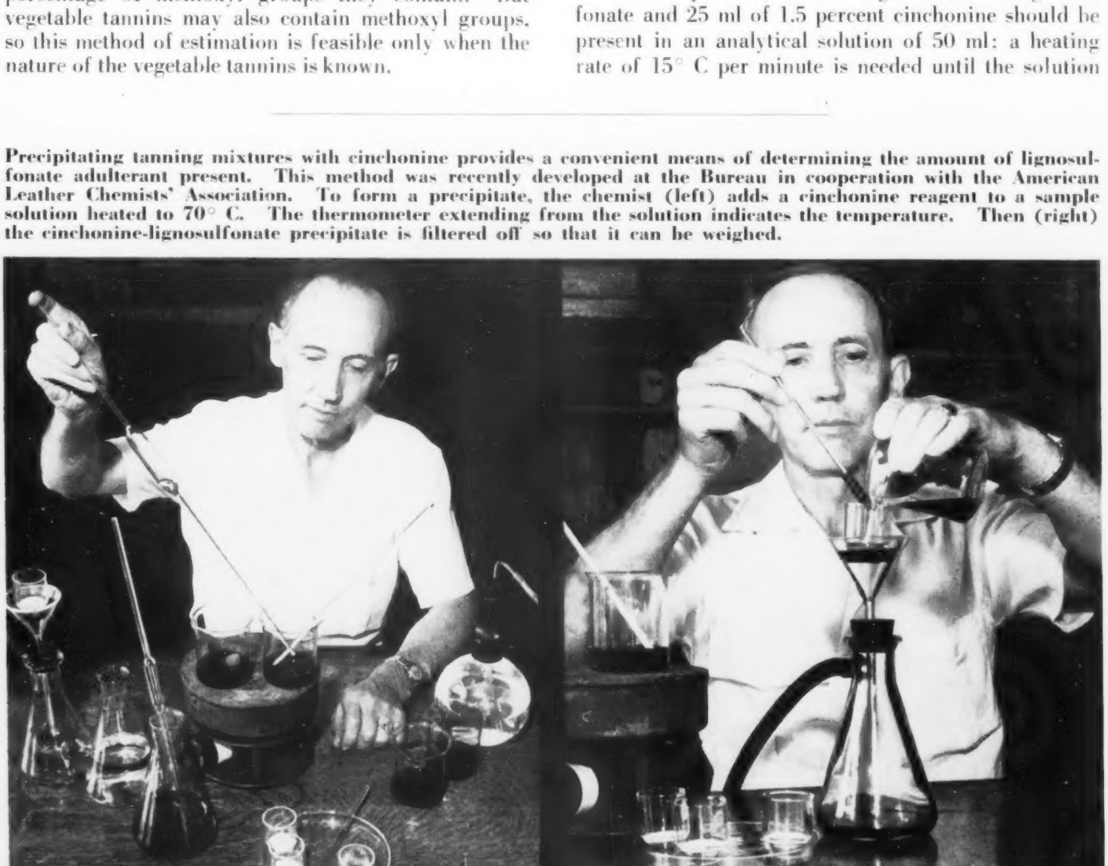
Estimating the amount of lignosulfonates in the presence of vegetable tannins is difficult as both mixtures have complex organic molecules with similar structures and functional groups. Lignosulfonates are characterized by their "methoxyl value," that is, the percentage of methoxyl groups they contain. But vegetable tannins may also contain methoxyl groups, so this method of estimation is feasible only when the nature of the vegetable tannins is known.

The method of precipitation of lignosulfonates with cinchonine was first suggested by Appelius and Schmidt.[2] According to their findings, lignosulfonates form insoluble compounds when a cinchonine reagent is added to a solution of vegetable tannins and hydrochloric acid. At elevated temperatures a lignosulfonate precipitate forms which can be filtered off, washed, dried, and weighed.

This method, highly empirical as described, was investigated by Sverre Dahl of the Bureau staff to determine its effectiveness. Evidence was available that the proper amount of hydrochloric acid was 5 ml per 100 ml of solution; that 2 min of boiling time was sufficient; that agitation should be avoided. Unknown factors were the character of tannin to be added to the solution; concentrations of lignosulfonates and of cinchonine; heating rate and temperature of precipitation; and drying time of the precipitate.

The NBS investigation found that the addition of tannic acid to the solution is essential, even though tannins are present; 0.1 to 0.4 gram of solid lignosulfonate and 25 ml of 1.5 percent cinchonine should be present in an analytical solution of 50 ml; a heating rate of 15° C per minute is needed until the solution

Precipitating tanning mixtures with cinchonine provides a convenient means of determining the amount of lignosulfonate adulterant present. This method was recently developed at the Bureau in cooperation with the American Leather Chemists' Association. To form a precipitate, the chemist (left) adds a cinchonine reagent to a sample solution heated to 70° C. The thermometer extending from the solution indicates the temperature. Then (right) the cinchonine-lignosulfonate precipitate is filtered off so that it can be weighed.



reaches a precipitation temperature of 70° C: proper drying time for the precipitate is 3 hours.[3]

These findings were confirmed by tests in the collaborating laboratories. A statistical analysis of the test results indicates that the cinchonine method can be considered serviceable under any conditions, even though the lignosulfonate concentration has not been chosen properly. The over-all absolute error was found to be ±3.84 percent lignosulfonate; this is fairly good accordance because lignosulfonates vary as to substance and chemical behavior. In the tests no precipitate was obtained when no lignosulfonate was present in the solution; also, the amount of precipitate had a definite relationship to the amount of lignosulfonate present.

Although not highly accurate or precise, the cinchonine method fills a need until a better method is developed. Studies of the problem are being continued by the American Leather Chemists' Association.

[1] *For further technical details, see* A collaborative study on the cinchonine method for determination of lignosulfonates in vegetable extract blends, *by Sverre Dahl and John Mandel, J. Am. Leather Chemists' Assoc.* **52**, *184 (1957)*.

[2] *W. Appelius and R. Schmidt, Collegium, 597 and 706 (1914)*.

[3] Report of the subcommittee for investigation of methods for detection and estimation of lignosulfonates in vegetable tannin blends, *by J. Jany, J. Am. Leather Chemists' Assoc.* **51**, *353 (1956)*.

1957 Conference on Bone Char Research

THE fifth biennial technical session on bone char research was held at the National Bureau of Standards, Washington, D. C., on May 16 and 17. Sponsored jointly by the U. S. Cane Sugar Refiners' Bone Char Research Project, Inc., and the Bureau, the conference was attended by members from Australia, Brazil, Canada, Cuba, England, and Scotland, as well as the United States. Sixteen technical papers summarizing accomplishments in bone char research were presented in the 2-day session.

The savory taste and purity of sugar depend on the adsorbent used in the refining process. As tremendous quantities are necessary to produce a first-rate sugar, the adsorbent must be capable of regeneration; that is, it must have a capability of being used over and over again to be economically successful. Several years ago a research program was started at the Bureau to study the fundamental nature of bone char, used as an adsorbent, in order to improve its effectiveness. Today industrial supporters of the program include sugar refiners and bone char manufacturers of nations throughout the free world.

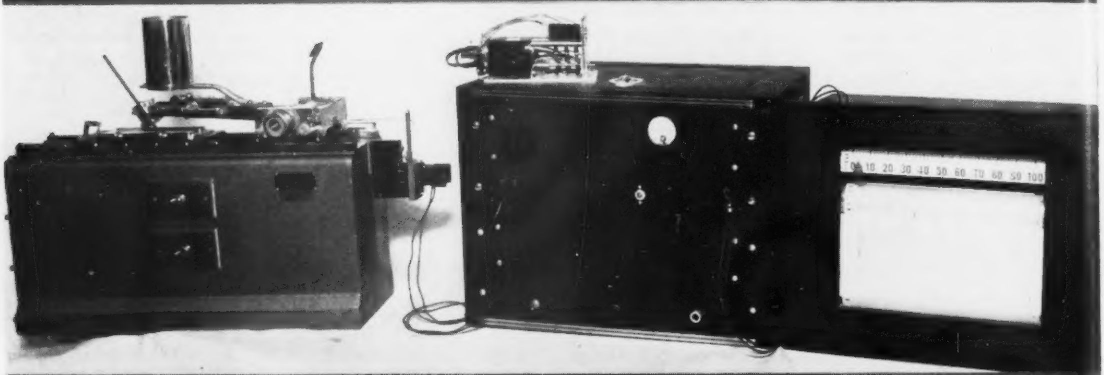
Conferees were welcomed at the opening meeting by Dr. A. V. Astin, Director of the Bureau. Thereafter, this meeting was devoted to the general topic of settling of char filters. Settling is a process by which regenerated bone char is combined with sugar liquor at the beginning of a filtration. Plant scale procedures for wet-settling were discussed, and the advantages of a method developed at the St. Lawrence Sugar Refineries were explained by E. J. Grant of Montreal. Results of research on the volume of sweet water per filter as a dependable criterion of settling techniques were given by H. G. Gerstner (Colonial Sugars Company, Gramercy, La.). A talk by Frank G. Carpenter of the Bureau staff concluded the first meeting. He presented a successful, new method for estimating the amount of sugar retained in an adsorbent after washing, by measuring the hydrogen and carbon monoxide produced during a heating process.

Problems in regeneration of bone char were discussed at the afternoon session. Laboratory tests at NBS for abrasion hardness were compared with refinery experiences. Inadequacies of some methods were clearly shown when chars of a known sequence were lined up in the wrong order. One paper gave the conclusions derived from a cooperative investigation undertaken by the National Sugar Refining Company (Philadelphia refinery) and Baugh and Sons Company (Philadelphia) on the effect of decarbonization of char. Results of a recent NBS study on the influence of excessive sulfate on the kilning behavior of bone char were discussed.

A highlight of the program was a talk on multiple treatments with different adsorbents by E. D. Gillette (Refined Syrups and Sugars, Inc., Yonkers, N. Y.) given the next morning. Plant scale experiences using bone char followed in sequence by granular carbon, powdered activated carbon, and ion exchange resins were described. The next paper by Kurt Löwy of Brazil proved that certain organic acids present in trace quantities in raw sugar liquors had a profound influence on the ability of bone char to decolorize the liquors.

Allen Gee, formerly of the Bureau staff, opened the final session Friday afternoon with a discussion of the anionic nature of the colorant in commercial sugar liquors. He proposed a hypothesis to explain the inferior initial performance of new char, namely, that new bone char has a property for rapidly exchanging divalent ions for monovalent ions. Victor R. Deitz of the Bureau introduced a novel concept to the effect that total organic anions in sugar liquor could be estimated by subtracting from total cations the sum of the equivalent of chloride and sulfate. Laboratory tests were compared to plant scale results for color removal by N. L. Pennington (California and Hawaiian Sugar Refining Corporation, Crockett, Calif.). The tests indicated that rough estimates of the performance of plant scale columns were possible. Similar tests for ash removal gave preliminary results which were even more promising. The final talk of the conference by William V. Loebenstein of the Bureau was a theoretical treatment of the adsorption of colorants during laboratory column filtrations.

THE INTEGROMETER



Improves Measurement of Adhesion

THE EVER-INCREASING SPEEDS of modern aircraft have developed a need for test methods to evaluate the adhesion property of protective coatings for these aircraft. So that potential coatings might be screened in the laboratory and only better types recommended for use in flight tests, the Bureau, under the sponsorship of the Navy Bureau of Aeronautics, recently made a study of available adhesion measuring instruments.

The adherometer is one of the promising instruments investigated. This apparatus measures the force required to strip a coating from a metal surface. A coating sample, mounted on a motor-driven plate, travels under a weighted knife. The variable stripping force encountered as the coating is removed is reflected on a dial gage. But this variable force is difficult to follow by dial gage readings; the process of recording and averaging many readings during a stripping run is lengthy and laborious; and results obtained are not always reliable.

To increase the speed, ease, and precision with which adherometer measurements could be made, A. G. Roberts and R. S. Pizer of the NBS plastics laboratory developed an electronic averaging device known as the Integrometer.[1] This device converts the variable stripping force measured by the adherometer into electric impulses. These impulses are then added to give a single average value which can be read directly from a standard recorder.

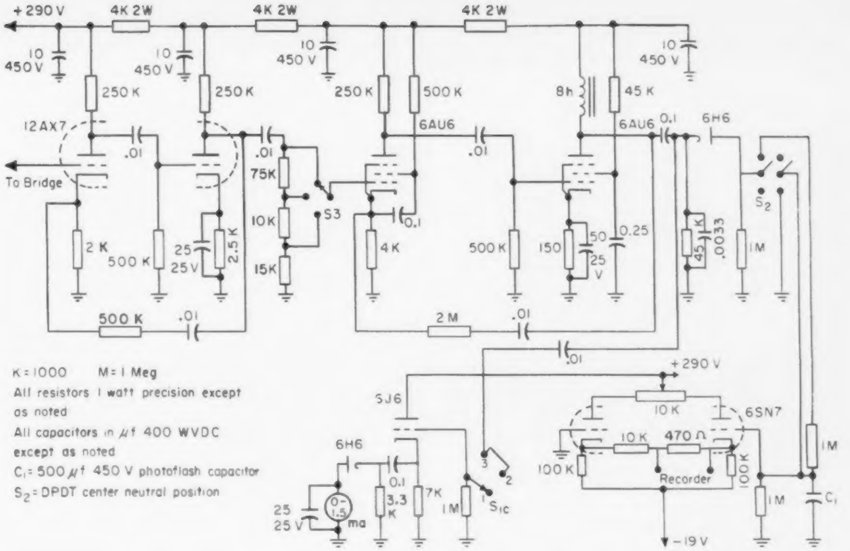
The first step was to modify the conventional adherometer by removing its pendulum and dial gage. The pendulum had been used as the source of the stripping force needed at the cutting head. An aluminum alloy beam with resistance strain gages attached to both faces was substituted for the pendulum. The Integrometer could then be used to perform the function of the dial gage.

The Integrometer contains a Wheatstone bridge circuit which includes the strain gages, an amplifier, integrating circuit, and meter circuit. Output from the beam and strain gage assembly is fed to the amplifier. The integrating circuit converts the amplified electric impulses to an output voltage proportional to the average value of the variable stripping force. The integrated voltage is then applied through the meter circuit to a standard recorder. The recorder draws a straight line the length of which, after a precisely timed interval, is proportional to the average value of the varying voltage that has been impressed on the integrating circuit during the stripping operation.

Thus the strain and error inherent in visually observing varying gage readings have been eliminated. Moreover, the stripping action itself is accomplished with greater ease and uniformity because the momentum and inertia effects of the pendulum method are removed.

Operation of the complete equipment has been simplified by the addition of a motor-driven switching assembly. At the push of a button, this assembly auto-

Above: The Integrometer, developed by the Bureau to improve adhesion measurements, is shown (center) connected to an adherometer (left) and to an automatic recorder (right). The adherometer measures the varying force required to strip a coating from its metal surface, as a coating sample, mounted on motor-driven plate, travels under a weighted knife. The Integrometer converts this variable stripping force into electric impulses and adds them to give an output that is indicated on the recorder.



Schematic diagram of amplifier, integrating circuit, and meter circuit of the Integrometer.

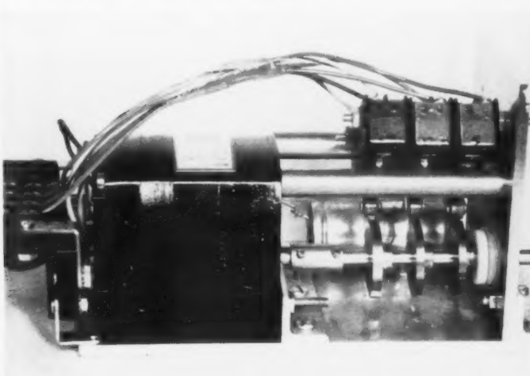
matically performs all of the timing and integrating operations necessary for a stripping run.

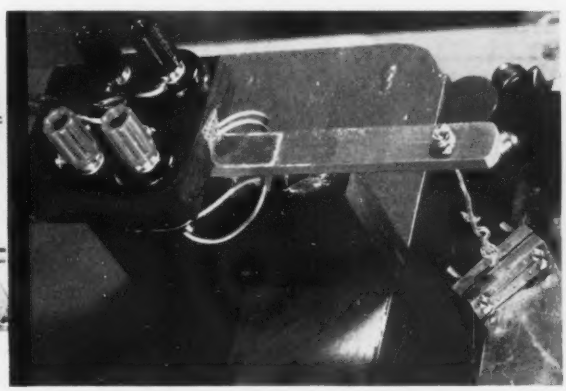
Stripping tests by the pendulum method on 11 different coating materials exhibited a coefficient of variation of 4.1 percent for replicate stripping runs. When the Integrometer was used, the coefficient of variation for replicate stripping runs was 2.2 percent. An analysis of the data indicated that the precision of the pendulum method varied from material to material and became poorer as the adhesion property of the coating became less uniform. The precision of the Integrometer method was independent of the material tested.

In addition to increasing the speed and precision of adherometer measurements, the Integrometer is expected to provide a more reliable experimental basis for interpretation of adherometer data. The device should also be useful in theoretical studies of adherometer operation and in correlation of the data with other adhesion measurements and service performance.

[1] *For further details, see* Protective coating adhesion measurement using an electronic averaging device for the adherometer, *by A. G. Roberts and R. S. Pizer, ASTM Bulletin No. 221, page 53 (1957).*

Below, left: **Closeup of the electronic switching assembly that simplifies operation of the Integrometer. This assembly automatically controls all of the timing and integrating operations for a stripping run. The three motor-driven cams actuate microswitches that control the various electronic circuits.** ***Right:*** **Closeup of aluminum beam and attached resistance strain gages which replace conventional pendulum and dial gage of adherometer. The beam is employed in place of pendulum as source of the stripping force needed at cutting head. The two strain gages form part of a Wheatstone bridge circuit inside the Integrometer. Bending of aluminum beam during a stripping run produces an electrical unbalance in the bridge.**





GASSING OF DRY CELLS

LECLANCHÉ CELLS—the dry cells commonly used in flashlights—have limited shelf life because of self-discharge through an internal chemical reaction. This deteriorating reaction produces gas which in time may bulge or even burst sealed batteries. Scientists at the Bureau have been investigating the production of gas in dry cells to find some relationship between the rate of gassing and battery life. The study was carried out for the Navy Bureau of Ships by E. M. Otto and W. G. Eicke, Jr., who constructed an apparatus that accurately measures the gassing rate of a single cell.

The chemical reaction that produces the gas—mostly hydrogen—when a cell is not in use is not the same one that produces useful current. Because the gaseous reaction promotes self-discharge with consequent deterioration of the cell, it has been assumed that cells with less tendency to evolve gas should have a longer shelf life. To test this assumption, a gas study technique seemed to be the most worthwhile approach. The Bureau therefore constructed equipment for accurately measuring the gassing rate. Experiments carried out with the aid of this apparatus showed that neither initial cell capacity nor retention capacity (capacity after a period of shelf storage) is a function of gassing rate. However, retention capacity does appear to depend on the total amount of gas evolved.

The gasometer consists essentially of four main parts in a closed system: (1) A container large enough to hold a size D cell, (2) a mercury reservoir, (3) a manometer, and (4) reference reservoir. Provision is made for removing gas samples during the run. The volume of gas produced can be computed from the amount of mercury withdrawn from the mercury reservoir to restore the pressure to its original value. Wires attached to the cell can be brought out through the container walls for closed circuit tests.

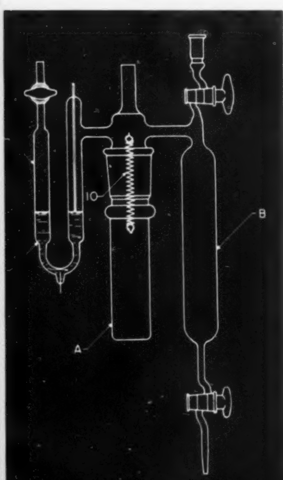
Runs were made at 21°, 35°, 45°, and 55° C because these temperatures have previously been used in shelf-life studies of dry cells. The tests were conducted when the cells were left on open circuit and when they were subjected to two different standard specified discharge rates. One of these discharges is intermittent through a 6.67-ohm resistor, and the other continuous through a 83.33-ohm resistor.

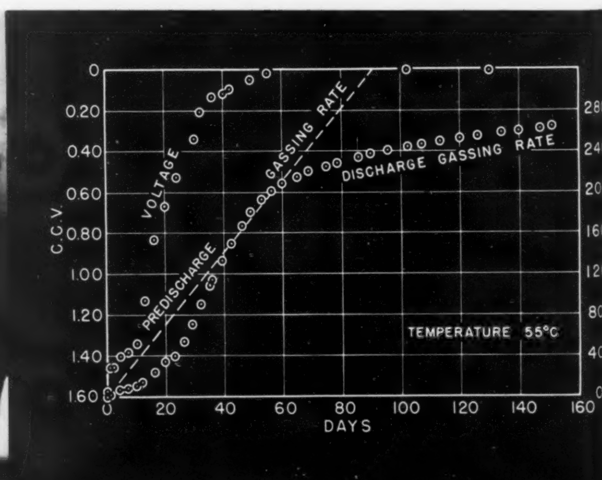
On open circuit, gassing proceeds at a nearly constant rate for any given temperature. The rate is greatly affected by temperature: for the values given above, the cells evolved respectively 0.1, 0.6, 2.0, 6.0 ml of gas per day. Conclusions from the electrical discharge tests are that evolution of a certain volume of gas during storage of a cell at high temperature is less detrimental than at low temperature. However, the time required to produce a given volume at higher temperatures is far less than at lower temperatures. For each temperature and type of cell, there appears to be a critical volume of gas a cell may evolve before there is a substantial loss in its capacity to generate current. Once the critical volume has been evolved, the cell deteriorates more rapidly.

It appears that no correlation exists between the rate of gassing and the initial capacity of the cell when the discharge test begins. When the cell is put on discharge, the immediate effect is to reduce the rate of gassing. Later, however, the rate increases, then tapers off again after the cell is exhausted. Gassing continues long after the cell has no useful electrical life.

For further technical information, see The gassing of dry cells, *by E. M. Otto and W. G. Eicke, Jr., J. Electrochem. Soc.* **104,** *199 (April 1957).*

Left: **Diagram of gasometer constructed by the Bureau to collect and measure gas in dry cell investigations. Principal parts are cell container (A), mercury reservoir (B), manometer (C), and reference reservoir (D).** ***Center:*** **Measuring the amount of gas produced by a dry cell with gasometer. The production of gas by dry cells has been under investigation to find some relationship between rate of gassing and battery life. These cells have limited shelf life because of self-discharge through an internal chemical reaction which produces gas.** ***Right:*** **Effect of continuous discharge on gassing of dry cells. Gassing rates can be found by determining the slope at any desired point on the curve.**





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SINCLAIR WEEKS, *Secretary*
NATIONAL BUREAU OF STANDARDS
A. V. ASTIN, *Director*

August 1957 Issued Monthly Vol. 41, No. 8

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